THE STATE OF CALIFORNIA

AIR RESOURCES BOARD

AIR MONITORING QUALITY ASSURANCE

VOLUME V

AUDIT PROCEDURES MANUAL

APPENDIX AB

SYSTEM AUDIT PROCEDURES
FOR DICHOTOMOUS PM10 SAMPLING AND ANALYSIS PROGRAMS

MONITORING AND LABORATORY DIVISION

MAY 1999

APPENDIX AB
SYSTEM AUDIT PROCEDURES FOR DICHOTOMOUS PM1O SAMPLING AND ANALYSIS PROGRAMS

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SYSTEM AUDIT PROCEDURES FOR DICHOTOMOUS PM10 SAMPLING AND ANALYSIS PROGRAMS

MONITORING AND LABORATORY DIVISION

MAY 1999

AB.1.0 SYSTEM AUDIT PROCEDURES FOR DICHOTOMOUS PM10 SAMPLING AND ANALYSIS PROGRAMS

AB.1.0.1 Introduction - A system audit is an on-site review and inspection of field sites and laboratory operations of a dichotomous PM10 (dichot) sampling program to assess its compliance with established regulations governing the collection, analysis, validation, and reporting of dichot sampling data. A system audit is normally conducted at the initiation of a new monitoring system and annually thereafter. A system audit includes an appraisal of the following program areas: network management, field and laboratory operations, data management and reporting, and quality assurance. On-site interviews should include a review of the data processing procedure from field acquisition through reporting into the information storage system (i.e., LIMS, AIRS).

The system audit is facilitated by the use of questionnaires designed to provide information about specific portions of the overall program. These questionnaires can be used together to provide a system audit of the whole program, or individually to provide a system audit on a portion of the program.

This procedure addresses the field and laboratory evaluations of a system and performance audit, including an evaluation of the field and laboratory operating procedures, mass analysis, and elemental analysis.

AB.1.0.2 Preliminary Assessment and System Audit Planning - In performing a system audit of a given district, the auditor is seeking a complete and accurate picture of that district's current dichot sampling operations. The auditor should perform the on-site inspections and interviews with key personnel, evaluate some dichot sampler sites operated by the district, and scrutinize the data processing procedures.

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GUIDELINES FOR CONDUCTING SYSTEM AUDITS

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AB.1.1 GUIDELINES FOR CONDUCTING SYSTEM AUDITS

A system audit should consist of three separate phases:
- Pre-Audit Activities
- On-site Audit Activities
- Post-Audit Activities.

AB.1.1.1 Pre-Audit Activities - At the beginning of each year, a tentative schedule for on-site system audits of the field sites and laboratories should be established. As part of this scheduling, the auditor should indicate any special requirements such as access to specific areas or observation of specific activities.

Approximately six weeks prior to the on-site audit, the auditor should arrange a tentative schedule for meetings with key personnel as well as for inspection of selected ambient air quality measurement and analytical operations. The auditor should also inform the district that they will receive a questionnaire which is to be completed and returned to the auditor within one month. Once the completed questionnaire has been returned, it will be reviewed, and the auditor will prepare a checklist detailing specific points for discussion with district personnel. The auditor should contact the district and coordinate the on-site audit.

AB.1.1.2 On-Site Audit Activities - The auditor should meet initially with the district's contact person or his or her designee to discuss the scope, duration, and activities involved with the audit. This should be followed by a meeting with key personnel identified from the completed questionnaire, or indicated by the district. Key personnel to be interviewed during the audit are those individuals with the responsibilities for: field and laboratory operations, data management and reporting, and quality assurance/quality control (QA/QC). The checklist of detailed specific points may be discussed during these meetings.

Enough time and effort should be devoted to the system audit so the auditor has a clear understanding and complete documentation in the following areas:

1. Organization
   - organization, training, and background of key personnel
   - general information on status of air monitoring program, QA plan, and field and lab Standard Operating Procedures (SOP)

2. Field Operations
   - conformance with regulations and QA/QC requirements
   - type of analyzers and samplers and conformance to 40 CFR Parts 53/58 requirements
   - field procedures, standards, documentation

- frequency of zero/span, calibration, precision
- corrective actions, repeat sampling runs
- standards certification, frequency, traceability
- spare parts, tools, records of repairs
- training as required or necessary
- data acquisition and handling reliability

3. Laboratory Operations
   - operational practices for manual methods
   - analytical methods used
   - use of SOP, QC use of duplicates and calibrations
   - corrective actions, repeat sample analysis
   - documentation and traceability for standards
   - record keeping, chain of custody, logbooks
   - waste disposal, safety practices, adequacy of lab for needs
   - data acquisition, data flow, back up, and validation

4. Data Management
   - data flow from field and lab to data processing
   - overview of data entry, automatic or manual
   - control check methods: if automatic, software and system
   - system backup and recovery capabilities
   - data screening, flagging, validation, correction (who may correct?)
   - type of reports, and responsibility for final validation

5. QA/QC Programs
   - status and implementation of procedures
   - outside audits
   - internal audits such as document reviews or data processing
   - implementation of corrective action
   - frequency, levels, and results of precision checks by pollutant

6. Reporting
   - precision and accuracy summaries
   - internal reports to track performance and corrective actions
   - summary of air data reports as required, completeness, and validity

In order to facilitate gaining a complete understanding of the dichot sampling and analysis program, the auditor should conduct a random spot check of the districts documentation and obtain sample copies of the following:

- Logs (daily calibration checks, weighings, maintenance, etc.)
- Calibration reports (dichot sampler, X-ray fluorescence (XRF), balance weights)

- Semi-annual relative humidity (RH) and temperature checks
- RH and temperature recordings
- Monthly report
- Quarterly QC report
- Dichot PM10 24-Hour Report Form (Figure AA.1.1.1), or equivalent
- Organization chart

Once the on-site system audit is complete, the auditor should meet again with key personnel and with the district's contact person or designee to present preliminary findings and possible recommendations. The auditor should state the audit results and include an indication of the potential data quality impact. This is also an opportunity for the district to present its disagreements.

The potential data quality impact is based upon specific criteria, some of which are requirements, and others which are only recommendations to improve the quality of a program. Specific criteria which must be met are found in 40 CFR Parts 50, 53, and 58, and in the "Quality Assurance Handbook for Air Pollution Measurement Systems", Volume II, Section 2.10 (see Reference #2). These criteria are summarized in Figure AA. 1.1.2.

AB.1.1.3 Post-Audit Activities - The major post-audit activity is the preparation of the System and Performance Audit Report. The preparation of this audit report requires the auditor to compare the documented standard operating procedures with the observed accomplishments and deficiencies of the audit findings.

If the deficiencies are such that the regulations and/or requirements are not met, then Air Quality Data Action requests (AQDAs) should be issued to the district. The AQDAs should note the pollutant, appropriate time period and reason for the issuance, as well as the time allowed for the district to respond.

The draft System and Performance Audit Report is submitted to the audited district, together with a letter requesting comments and thanking district personnel for their assistance, time, and cooperation. If no written comments are received within 30 calendar days from the report date, the report will be formally distributed without further changes.

If the district has written comments or questions concerning the audit report, they should be reviewed for incorporation into a final report within 30 days of receipt of the written comment.

The System and Performance Audit Report should include the following:
- an executive Summary
- an introduction
- audit results based on the questionnaire responses and on-site observations

- a discussion of the interpretation of the audit results
- recommendations for improving the program,
- timeline for implementing the recommendations
- follow-up items
- a copy of the completed questionnaire

The audit results should include information on the staff and equipment, on the network size and siting criteria, on the data management system, on the quality assurance and quality control functions, and on AQDAS issued, if any, including resolution of such AQDAS.

DICHOTOMOUS PM10
24 - HOUR REPORT FORM

Bar Code:

LIMS Sample ID:

Station Name:

Elevation:

Reporting Agency:

Station Address:

Station Operator:

Operator Phone #:

EPA AIRS SITE # State County Site

Instrument

Collocated?

Sample Date: Year Month Day

Sampling Temperature and Pressure:

Ambient Temp. °C

Ambient Presssure mmHg

A: No Unusual Conditions
B: Wind - Blown Dust/Sand
C: Construction Nearby
D: Farming Nearby
E: Fire Nearby
H: Rain
I: Snow
Z: Other_____
Sampling Condition

Date Of Calibration: Year Month Day

LAB USE ONLY

Rotometer Reading: Coarse
Initial:
Final:
Average:

Rotometer Reading: Total
Initial:
Final:
Average:

Local (Actual) Flow LPM

Elapsed Time Meter
Final: min
Initial: min
Net: min

Determination of Standard Flow Rate in SLPM:

Rotometer Reading Average (Total Flow) X Slope (Total Flow) ± Intercept (Total Flow) = Total Flow in SLPM

Comments:

FOR LABORATORY USE ONLY:

	Coarse	Fine	CDUP	FDUP
Post-weight:				
Pre-weight:				

Received at ARB Lab on:

Pre-wt. Date: Post-wt. Date: Remarks:

Pre-wt. Analyst: Post-wt. Analyst:

REV. 8/98

Figure AB.1.1.1
Dichotomous PM10 24-Hour Report Form

QUALITY CONTROL CRITERIA FOR
DICHOTOMOUS (PM10) SAMPLING AND ANALYSIS

1. Filters
   a. The filters must meet the criteria outlined in 40 CFR Part 50, Appendix J with respect to physical characteristics. The district conducting dichot sampling must adhere to the following practices:
      1) filters must have unique identification numbers.
      2) filters must be equilibrated in a controlled environment for a minimum of 24 hours under the following criteria prior to pre- and post-run weighing:
         (a) Temperature range: 15°C to 30°C
         (b) Temperature control: ±3°C
         (c) Relative Humidity range: 20% to 45%
         (d) Relative Humidity control: ±5%
      3) The relative humidity (RH) and temperature must be recorded on equilibration days.
      4) A minimum of 10% of all filters must be re-weighed.

2. Instruments
   a. The analytical balance must have a minimum sensitivity of ±1 microgram (ug), and accuracy of ±4ug at zero and ±2ug at 10 milligrams (mg). A minimum of American Society for Testing and Materials (ASTM) Class 1 weights are to be used for calibrations.
   b. A thermometer capable of measuring ambient temperature over a range of 10 to 30 °C to the nearest ±0.1°C. This temperature sensor should be traceable with an accuracy of 0.1 °C to a National Institute of Standards and Technology (NIST) certified thermometer of an American Society for Testing and Materials thermometer (ASTM) thermometer within ±0.1°C.
   c. The RH sensor must be referenced every 6 months to a wet/dry bulb psychrometer within +6%.
   d. The barometer must be capable of measuring barometric pressure over a range of 500 to 800 mm Hg to the nearest mm of Hg and be referenced annually to a standard within ±5 mm Hg.
   e. The dichot sampler must meet U.S. EPA operational standards and be a model designated as a reference or equivalent method and must be operated according to the following criteria:
      1) tolerance of ±10% of the design inlet flow rate, i.e., total flow=16.7 L/min ±10% and coarse flow=l.67 L/min ±10%
      2) calibration with a certified transfer standard at least annually within ±4%; recalibration if difference is greater than ±7% during calibration checks or audits
      3) difference between true flow and indicated flow must be no greater than ±10%
      4) average sampling run flow rate must be within ±7% of design
      5) the sampling run time must begin and end within 1/2 hour of midnight and must be at least 23 hours but no longer than 25 hours (1380 to 1500 minutes).

Figure AB.1.1.2
Dichotomous Sampling and Analysis Quality Control Criteria

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CRITERIA FOR EVALUATION

MONITORING AND LABORATORY DIVISION

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AB.2.0 CRITERIA FOR EVALUATION

AB.2.0.1 Introduction - A system audit is normally conducted in three steps. First, a questionnaire is sent to the district prior to the audit visit. The district should fill out the questionnaire as completely as possible and return it with sufficient documentation through the use of attachments. Second, the questionnaire is reviewed by the auditor to become familiar with the system operations and to determine any weaknesses and potential problem areas. Third, the on-site visit and interviews are scheduled.

For the field audit, the auditor should interview the site operator. For the laboratory audit, the auditor should interview the laboratory manager, any person who has direct analytical responsibility for dichot sampling analysis, personnel associated with data validation, analysis and reporting, and the person identified by the laboratory manager who has responsibility for quality assurance. The information gathered from these interviews should be complete and up-to-date. It also should present an adequate picture of the current and proposed levels of implementation of all quality assurance activities, including internal quality control. An evaluation of the data handling and processing, field and laboratory operations and procedures, QA/QC, and analytical process should be conducted at this time.

At the conclusion of the series of interviews and the evaluations, the auditor should inform the district contact person of the audit results and discuss any potential data-impacting problems uncovered. At this time, the auditor also explains the reporting procedures and schedule.

AB.2.0.2 Questionnaire - An overall program operations questionnaire is intended for use when a complete system audit is being conducted in conjunction with the field/and or laboratory operations questionnaires. The overall program operations questionnaire should be completed by the person responsible for the overall program and should be returned to the auditor.

The questionnaire includes several areas including the reporting organization homogeneity, general operation, staffing, network design, network operation, data and record keeping, and quality assurance. This questionnaire is intended to cover the management and organizational activities of the program.

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CRITERIA FOR EVALUATION

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AB.2.1 DICHOTOMOUS SYSTEM AUDIT QUESTIONNAIRE

Agency_____

Address_____

Phone Number (___) _____

Organization Director_____

Dichot Program Supervisor_____

Data Management Supervisor_____

Quality Assurance Officer_____

Questionnaire Completed_____
(By) (Date)

On-Site Visit
Date _____ Audit Team Members_____

Affiliation of Audit Team_____

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A. REPORTING ORGANIZATION HOMOGENEITY

1. Reporting Organization Homogeneity

a. Are field operations conducted by a common team of field operators? Yes[ ] No[ ]

b. Are common calibration facilities used for all sites and laboratories? Yes[ ] No[ ]

c. Are precision checks performed by common staff for all sites and laboratories? Yes[ ] No[ ]

d. Are accuracy checks performed by common staff for all sites and laboratories? Yes[ ] No[ ]

e. Are uniform procedures followed for data handling at all sites and laboratories? Yes[ ] No[ ]

f. Is a central data processing facility used for all reporting? Yes[ ] No[ ]

g. Is the traceability of all standards established by one central support laboratory? Yes[ ] No[ ]
Who operates this lab?_____

h. Does one central analytical laboratory handle all analyses for manual methods? Yes[ ] No[ ]
Who operates this lab?_____

B. GENERAL OPERATIONS

1. Dichot Sampling Program

a. When was the dichot sampling program initiated?_____

b. How many sites are operated?_____

c. Please complete the following information:

Site	AIRS Site #	Operating Since	Collocated Sampler(Yes or No)

B. GENERAL OPERATIONS (cont.)

2. Standard Operating Procedures (SOP)

a. Have the following been developed:

Quality Assurance Project Plan? Yes[ ] No[ ]
Date _____

Documentation on sites and network? Yes[ ] No[ ]
Date _____

Standard Operating Procedures for Field Operations? Yes[ ] No[ ]
Date _____

Standard Operating Procedures for Analytical Laboratory? Yes[ ] No[ ]
Date _____

Laboratory Quality Control Manual? Yes[ ] No[ ]
Date _____

b. Have the documents listed above been provided to the Quality Assurance Section? Yes[ ] No[ ]

c. Does the program operate in compliance with:
U.S. EPA protocol? Yes[ ] No[ ]
ARB protocol? Yes[ ] No[ ]

3. Staffing

a. Provide a current organization chart indicating each responsible person's role in the current program.

b. Please include a list of educational background, experience and training for each responsible person identified in the program organization chart.

C. NETWORK

1. Design

a. Are all sites documented according to specified criteria? Yes[ ] No[ ]

C. NETWORK (cont.)

b. Has the network been designed in accordance with the stated program objectives? Yes[ ] No[ ]
Please include a brief description of any siting compromises.

c. Is there a written plan describing the overall network? Yes[ ] No[ ]
Title_____
Date_____

d. Does the organization have records identifying the status and history of each site, which includes:
1) site identification? Yes[ ] No[ ]
2) site coordinates and elevation? Yes[ ] No[ ]
3) date monitoring initiated? Yes[ ] No[ ]
4) model, manufacturer and serial numbers of equipment at the site and sampling schedule? Yes[ ] No[ ]
5) reason for periods of missing data? Yes[ ] No[ ]

e. Is equipment at all sites installed in accordance with:
1) manufacturer's specifications? Yes[ ] No[ ]
2) network guidelines? Yes[ ] No[ ]
3) sound scientific principles? Yes[ ] No[ ]

f. Does the network design consider:
1) access? Yes[ ] No[ ]
2) power availability? Yes[ ] No[ ]
3) potential localized interferences such as closely located sources? Yes[ ] No[ ]

g. How often are sites visited by the primary operator?_____

h. How often are the samples removed?_____

2. Operation

a. Is equipment in the network operated in accordance with the organization's standard operating procedures (where such exist)? Yes[ ] No[ ]

b. Are the operating procedures compatible with:
1) U.S. EPA guidelines? Yes[ ] No[ ]
2) manufacturer's recommendations? Yes[ ] No[ ]
3) ARB's Air Monitoring Quality Assurance SOP? Yes[ ] No[ ]

C. NETWORK (cont.)

c. Is equipment operated on a documented schedule? Yes[ ] No[ ]
(Please attach a copy)

d. Are an adequate supply of spare parts and expendables maintained at all sites? Yes[ ] No[ ]

e. Are all sites operated year round? Yes[ ] No[ ]

f. Is a bound logbook maintained at all sites? Yes[ ] No[ ]

Does the logbook include:
1) records of all site visits? Yes[ ] No[ ]
2) problems and repairs? Yes[ ] No[ ]
3) maintenance? Yes[ ] No[ ]
4) data? Yes[ ] No[ ]

g. Is routine maintenance performed at all sites? Yes[ ] No[ ]
By whom?_____
(Name and position)

h. Does the person performing such maintenance have access to standard troubleshooting/maintenance procedures or instrument manuals? Yes[ ] No[ ]

I. Are any measurements made on samples at sites? Yes[ ] No[ ]
If so, please describe these measurements.

j. Please describe how samples are shipped or transported to the analytical laboratory.

D. DATA AND RECORD KEEPING

1. Data Handling

a. Please indicate the data that is maintained and the data format:
   1) field site data
      (i) calibration data_____
      (ii) sampling run data_____
   2) field lab data_____
   3) analytical lab data:
      (i) analytical results_____
      (ii) calibration data _____
      (iii) separate QC data _____

b. Are field data checked for reasonableness? Yes[ ] No[ ]

c. Are analytical lab data checked for reasonableness? Yes[ ] No[ ]

d. Are a portion of the data from the field verified by the lab (such as collocated sampling)? Yes[ ] No[ ]

e. Are replicate results tabulated and available for review? Yes[ ] No[ ]

f. Are cross-checks used to validate or flag data? Yes[ ] No[ ]

g. Please describe what corrective actions are taken for out-of-control situations.

h. Please describe how data are adjusted or deleted for out-of-control situations.

2. Reporting

a. In what format are results reported (e.g., hard copy, diskette, electronically)?_____

b. To which agency are the results reported (e.g., U.S. EPA, ARB, etc.)?
_____

D. DATA AND RECORD KEEPING (cont.)

c. To whom specifically are the results reported (Name of Person and Division/Section Title)?_____

d. How often are the results forwarded to the reporting organization?

_____

e. Where and how are data stored? For how long?_____

_____

E. QUALITY ASSURANCE

1. Quality Assurance

a. Is there a defined quality assurance function ongoing within the network? Yes[ ] No[ ]

b. Is this function independent of all routine operations? Yes[ ] No[ ]

c. Does the individual responsible for this function regularly evaluate or audit the following operations:

1) site operations (performance audits)? Yes[ ] No[ ]
2) site data? Yes[ ] No[ ]
3) analytical laboratory operations? Yes[ ] No[ ]
4) analytical laboratory data? Yes[ ] No[ ]

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APPENDIX AB.3.0

FIELD OPERATIONS EVALUATION

MONITORING AND LABORATORY DIVISION

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AB.3.0 FIELD OPERATIONS EVALUATION

AB.3.0.1 Introduction - A field operations system audit follows the procedures outlined in Section AB.2.0.1, Criteria for Evaluation. The system audit consists of 3 steps: 1) sending a questionnaire to the district prior to the audit visit, 2) reviewing the completed questionnaire, and 3) conducting the on-site visit and interviews. It may be necessary to visit one or more of the air monitoring sites. Therefore, it is highly recommended that arrangements with the district be made in advance of the on-site visit.

During the on-site visit, the auditor should interview the site operator responsible for the dichot sampler, personnel associated with field data validation, analysis, and reporting, and the person identified who has responsibility for quality assurance. The information gathered from these interviews should be accurate, and should present an adequate picture of the current and proposed levels of implementation of all quality assurance activities, including internal quality control. An evaluation of the data handling and processing, field operations and procedures, and QA/QC should be conducted at this time. This evaluation should consist of, at a minimum, a random verification of district records.

At the conclusion of the series of interviews and evaluations, the auditor should inform the district contact person of the audit results and discuss any potential data-impacting problems uncovered. At this time, the auditor also explains the reporting procedures and schedule.

AB .3.0.2 General Guidance for Site Documentation - During the initial phase of network installation, each site should be documented using a site report form. This form should be completed by organization personnel to record station location, site classification, station instrumentation, topography and important pollution sources. This documentation should be updated at least annually thereafter, to reflect the changes that occur at the sites (e.g., construction of a new building).

It is important that the information contained on such site documentation be verified as accurate. While it does not fall within the scope of the quality assurance function to prepare these site documents, the auditor should verify, for a small number of sites, that the information contained in such documents is accurate and complete. He/she should note any changes which may affect data quality and notify organization management of such problems. Of particular importance in this regard are sites where collocated instrumentation has been placed, such data may be used to estimate measurement or data precision.

AB.3.0.3 Site Evaluation Reporting - At the conclusion of a site evaluation or evaluation of a group of sites for a single organization, the auditor should prepare a brief written report (refer to Section AA.1.1.3). This report should include at least a discussion of observations made during the site visit as noted in the questionnaire and a copy of the site documentation used for the evaluation. Where major discrepancies are noted, additional information needs to be included. If further documentation has been provided by the auditor, a newly completed accurate site description document should be attached. Recommendations to improve siting should be included.

AB.3.0.4 Questionnaire - A field operations system audit questionnaire should be completed by every person involved in sample and data handling, operations of a field site and field activities quality control. The completed questionnaire will provide information on site documentation and field site evaluation.

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FIELD OPERATIONS SYSTEM AUDIT QUESTIONNAIRE

MONITORING AND LABORATORY DIVISION

MAY 1999

AB.3.1 FIELD OPERATIONS SYSTEM AUDIT QUESTIONNAIRE

Agency_____

Address_____

Phone Number (___)_____

Organization Director_____

Dichot Program Supervisor_____

Data Management Supervisor_____

Quality Assurance Officer_____

Questionnaire Completed_____
(By) (Date)

On-Site Visit
Date_____ Audit Team Members_____

Affiliation of Audit Team_____

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A. SITE DOCUMENTATION

1. Site Location

a. Please provide the following information for each dichot sampling site:

SITE	ID NUMBER	ADDRESS	LONGITUDE AND LATITUDE	IS DOCUMENTATION CURRENT?

b. Is there an up-to-date map indicating location and distances to the major sources which may impact the data gathered at the site? Yes[ ] No[ ]

c. Does this site map include indications of roadways, parking areas, buildings, tree lines, power lines, bodies of water, and fences? Yes[ ] No[ ]

2. Equipment

a. Is all instrumentation present and operational? Yes[ ] No[ ]
If no, please explain the reason for the non-operation, estimate the down time, and indicate whether this is a potential data quality impactor.

b. Has additional equipment been added since the site documentation was prepared, or has equipment been removed or changed? Yes[ ] No[ ]
Please note such equipment and identification numbers.

c. For each dichot sampler site:
   1) Does each sampler have unobstructed air flow for a minimum of 2 meters (m) in all directions? Yes[ ] No[ ]
   2) Is each sampler inlet placed at a height of 2 to 15 m above ground level? Yes[ ] No[ ]

A. SITE DOCUMENTATION (cont.)

3) Is the sampler located away from obstacles such as buildings, so that the distance between obstacles and the sampler is at least twice the height that the obstacle extends above the sampler? Yes[ ] No[ ]

4) If collocated with another particulate matter sampler, is there a minimum spacing of 2 m between sampler inlets and a maximum spacing of 4 m? Yes[ ] No[ ]

5) If collocated with another PM10 sampler, are the sampler inlet heights within 1 vertical meter of one another? Yes[ ] No[ ]

B. FIELD SITE EVALUATION

1. Operations

a. Field Activities Standard Operating Procedures (SOP)

1) Has the District developed and implemented a SOP? Yes[ ] No[ ]
2) Is the SOP manual followed in detail? Yes[ ] No[ ]
3) Does the SOP contain all quality control steps practiced? Yes[ ] No[ ]
4) Does each site operator have a copy at his/her disposal? Yes[ ] No[ ]

b. Is a formal training program used for site operators? Yes[ ] No[ ]

2. Calibrations

a. Does the District have the necessary hand tools, electrical testing and calibration equipment to operate and maintain equipment, calibrate dichot sampler flow controllers, and repair samplers at each site? Yes[ ] No[ ]

b. How often is each dichot sampler calibrated?

_____

c. Is each sampler calibrated:

1) after maintenance? Yes[ ] No[ ]
2) any time audits or flow checks deviate more than ±7% from the indicated flow rate? Yes[ ] No[ ]
3) any time audits or flow checks deviate more than ±10% from the design flow rate? Yes[ ] No[ ]

d. Is a transfer standard, that is traceable to a NIST primary standard, used for calibration of each sampler? Yes[ ] No[ ]

e. Is the flow-rate transfer standard checked against a NIST primary standard annually? Yes[ ] No[ ]

B. FIELD SITE EVALUATION (cont.)

f. Is a field calibration check of the total and coarse flow rates conducted on each sampler after every month of operation? Yes[ ] No[ ]

g. Is the following equipment and information used for field calibration checks:

1) thermometer capable of accurately measuring temperature to the nearest ±1 °C and referenced annually to an NIST or AST thermometer within ±2 °C? Yes[ ] No[ ]

2) barometer capable of accurately measuring ambient barometric pressure to the nearest ±1 mm Hg and referenced annually to a NIST or ASTM barometer within ±5 mm Hg? Yes[ ] No[ ]

3) two calibrated orifice devices (one for total and one for coarse flow) and calibration relationships? Yes[ ] No[ ]

4) sampler's calibration information? Yes[ ] No[ ]

5) two clean flow-check filters? Yes[ ] No[ ]

6) dichot sampler flow-check data sheet or log book? Yes[ ] No[ ]

h. Are the calibration and calibration check data recorded in a log book or sheet? Yes[ ] No[ ]

i. Is the sampler timer accurate to within ±2 minutes/24 hours? Yes[ ] No[ ]

3. Filter Handling

a. Are filters visually inspected for damage, loose materials, poor workmanship, or irregularities? Yes[ ] No[ ]

b. Does each filter have a unique identification number assigned? Yes[ ] No[ ]

c Please describe how filters are transported.

4. Documentation

a. Does a Dichot PM10 24-Hour Report Form, or equivalent, accompany each filter at all times? Yes[ ] No[ ]

b. Is a log book/log sheet maintained at the site? Yes[ ] No[ ]

c. Is a maintenance log book for the dichot sampler maintained at the site? Yes[ ] No[ ]

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LABORATORY OPERATIONS EVALUATION

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AB.4.0 LABORATORY OPERATIONS EVALUATION

AB.4.0.1 Procedure - A laboratory system audit follows the procedures outlined in Section AB.2.0.1, Criteria for Evaluation. That is, the system audit is conducted in three steps: 1) a questionnaire is sent to the analytical laboratory prior to the audit visit, 2) the questionnaire is reviewed by the auditor, and 3) the on-site visit and interviews are scheduled.

During the on-site visit, the auditor should interview the laboratory manager, any person who has direct analytical responsibility for dichot sampling analysis, personnel associated with data validation, analysis and reporting, and the person identified by the laboratory manager who has responsibility for quality assurance. The information gathered from these interviews, complete and up-to-date, should present an adequate picture of the current and proposed levels of implementation of all quality assurance activities, including internal quality control. An evaluation of the data handling and processing, laboratory operations and procedures, QA/QC, and analytical process should be conducted at this time.

At the conclusion of the series of interviews and evaluations, the auditor should inform the laboratory manager of the audit results and discuss any potential data impacting problems uncovered. At this time, the auditor also explains the reporting procedures and schedule.

AB.4.0.2 Questionnaire - A laboratory Questionnaire provides information on analytical methods, standard laboratory operations, data entry, data bank validation, laboratory quality control, and laboratory management. The laboratory system audit questionnaire should be completed by every person involved in the data entry and review process, and by every person responsible for the operation of an analytical instrument.

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VOLUME V

AUDIT PROCEDURES MANUAL

APPENDIX AB.4.1

LABORATORY OPERATIONS SYSTEM AUDIT QUESTIONNAIRE

MONITORING AND LABORATORY DIVISION

MAY 1999

AB.4.1 DICHOTOMOUS LABORATORY OPERATIONS SYSTEM AUDIT QUESTIONNAIRE

Agency_____

Address_____

Phone Number (____)_____

Organization Director_____

Dichot Program Supervisor_____

Data Management Supervisor_____

Quality Assurance Officer_____

Questionnaire Completed_____
(By) (Date)

On-Site Visit
Date_____ Audit Team Members_____

Affiliation of Audit Team_____

TABLE OF CONTENTS

A. GENERAL OPERATIONS

1. Dichot Sampling Program

   a. How long has the district been operating a dichot sampling program?

   _____

   b. How many sites are operated?_____

   c. Please list the dichot sampling sites operated by the District.

Site	AIRS Site #	Operating Since	Collocated Sampler (Y or N)

2. Sampling Analysis

   a. How long has the District been conducting mass analysis for the Dichot Sampling Program?_____

   b. How long has the District been conducting elemental analysis for this program?

   _____

B. STAFFING

1. Staffing

   a. Provide a current organization chart indicating each responsible person's role in the current program.

   b. Please include a list of educational background, experience and training for each responsible person identified in the program organization chart.

   c. Are staff members adequately conversant with appropriate standard operating procedures to carry out job duties? Yes[ ] No[ ]

   d. If there is not a Quality Assurance Officer, who handles this responsibility?_____
   (Name)
   To whom does he/she report?_____
   (Name and Title)

C. EQUIPMENT AND ENVIRONMENT

1. Standard Operating Procedures (SOP)

   a. Are standard operating procedures available for filter processing, weighing, and elemental analysis? Yes[ ] No[ ]

   b. Does the SOP include the chain of custody document? Yes[ ] No[ ]

2. Calibration Weights

   a. Are American Society for Testing and Materials (ASTM) class 1 or better weights used? Yes[ ] No[ ]

   b. If so, are they weighed to the nearest 0.001 mg? Yes[ ] No[ ]

   c. Record the actual readings obtained using your class 1 total weights:

   1)_____ 2)_____ 3)_____

   4)_____ 5)_____ 6)_____

3. Microbalance

   a. Is a serial/property number assigned? Yes[ ] No[ ]
   (#_____)

   b. Do microbalance specifications have:
   1) Resolution to 0.001 mg? Yes[ ] No[ ]
   2) Precision of ±0.001 mg? Yes[ ] No[ ]

   c. Is the microbalance calibrated annually? Yes[ ] No[ ]
   Date of last calibration?_____

4. Filter Equilibration

   a. Are filters equilibrated for a minimum of 24 hours in a controlled environment before weighing? Yes[ ] No[ ]

   b. Temperature
   1) Is the temperature held constant (±3 °C) between 15 °C and 30 °C? Yes[ ] No[ ]
   2) Is the thermometer checked and temperature continually recorded on equilibration days? Yes[ ] No[ ]
   3) If the temperature is checked manually, how often is it recorded? _____

C. EQUIPMENT AND ENVIRONMENT (cont.)

4) Is the thermometer checked semi-annually against a reference thermometer? Yes[ ] No[ ]

c. Relative Humidity (RH)

1) Is the RH held constant (±5%) between 20% and 45%? Yes[ ] No[ ]

2) Is the RH instrument checked and RH continually recorded on equilibration days? Yes[ ] No[ ]

3) If the RH is checked manually, how often is it recorded?

_____

4) Is the RH instrument checked semi-annually against a sling psychrometer or other reference RH meter? Yes[ ] No[ ]

5. X-Ray Fluorescence Analyzer

a. Does the district conduct elemental analysis on the dichot sampling filters? Yes[ ] No[ ]

b. If the District does not analyze the dichot filters, are the filters analyzed by the ARB? Yes[ ] No[ ]

c. Are the filters analyzed by some other lab? Yes[ ] No[ ]
If so, which lab?_____

d. If the district analyzes the dichot filters, does the district analyze using X-ray fluorescence (XRF)? Yes[ ] No[ ]
If not by XRF, by what method is elemental analysis conducted?_____

e. Is the XRF Analyzer recalibrated:

1) every 3 months? Yes[ ] No[ ]

2) when the quality assurance standards indicate a drift of more than ±3% in calibration? Yes[ ] No[ ]

3) when the results of the QA standard, if analyzed in every run, consistently fall outside the ±5% intervals? Yes[ ] No[ ]
Date of last calibration?_____

f. While conducting calibrations, are 2 to 4 blanks analyzed in the first run? Yes[ ] No[ ]

g. What standards are used for calibration?

1) elemental thin film Yes[ ] No[ ]

2) multiple element thin film Yes[ ] No[ ]

3) NIST certified thin film Yes[ ] No[ ]

Other:_____

C. EQUIPMENT AND ENVIRONMENT (cont.)

h. Are all standards verified for accuracy prior to use for calibration? Yes[ ] No[ ]

i. After every instance in which the electrical power to the Multi-Channel Analyzer (MCA) is restored, is the MCA calibrated for the properly labeled energy channels? Yes[ ] No[ ]

j. Is the energy calibration of the MCA gauged using a Kevex stainless steel disk and using Fe K alpha line at 6.400eV and the Mo K alpha line at 17.443eV? Yes[ ] No[ ]

k. Describe what action is taken if the energy calibration accuracy exceeds ± 3 eV based on Fe and Mo peaks in a Kevex stainless steel standard.

6. Filter Handling

a. Are gloves worn while handling the filters? Yes[ ] No[ ]

b. Are non-serrated forceps used to handle filters? Yes[ ] No[ ]

D. PRE-RUN FILTER INSPECTION AND WEIGHING

1. Filters

a. What type and size of filters are used? _____

b. Where are the filters obtained? (EPA, ARB, etc.)

_____

c. If from the ARB, are the filters delivered pre-weighed? Yes[ ] No[ ]

d. Are acceptance tests performed on all filter manufacturer lots? Yes[ ] No[ ]

e. Are the filters visually inspected for damage, pinholes, seal integrity, sagging, puckering, loose materials, poor workmanship, discoloration, non-uniformity, or irregularities? Yes[ ] No[ ]

f. Are filters numbered sequentially and listed in a bound laboratory log book or computer? Yes[ ] No[ ]

g. Are filters outside the 80 to 110 mg range rejected or investigated? Yes[ ] No[ ]

h. Are filters stored in protective petri dishes? Yes[ ] No[ ]

D. PRE-RUN FILTER INSPECTION AND WEIGHING (cont.)

i. If the filters are mailed, are they sufficiently protected in the mailing envelope? Yes[ ] No[ ]

2. Log Books/QC Check Sheets

a. Is a Log Book/Log Sheet maintained? Yes[ ] No[ ]

b. Are the Mass Analysis data maintained in a separate log book/ log sheet than the XRF Analysis data? Yes[ ] No[ ]

c. Is an XRF sample analysis list maintained? Yes[ ] No[ ]

d. Is a maintenance log book maintained for all laboratory equipment? Yes[ ] No[ ]

e. Are all log books/log sheets current? Yes[ ] No[ ]

f. Are they legible? Yes[ ] No[ ]

g. Do they show calibrations? Yes[ ] No[ ]

h. Are they initialed by the operator? Yes[ ] No[ ]

i. Are they dated? Yes[ ] No[ ]

j. Do they show filter weights? Yes[ ] No[ ]

k. Is the data archived? Yes[ ] No[ ]
If so, for how long is the data stored?_____

3. Microbalance

a. Is a microbalance internal calibration performed before each weighing session? Yes[ ] No[ ]

b. Is the microbalance zero value checked after each reading And rezeroed as needed? Yes[ ] No[ ]

c. Is a 100-mg calibration check of the microbalance performed after every ten or less weighings? Yes[ ] No[ ]

D. PRE-RUN FILTER INSPECTION AND WEIGHING (cont.)

d. Describe what action is taken if the zero and calibration checks exceed the acceptable limits (electronic zero within ±0.004 mg and calibration within ±0.002 mg).

e. After completion of the zero and calibration checks, is one arbitrarily-selected filter from a set of "standard" filters tare weighed? Yes[ ] No[ ]

f. Are tare weights checked by reweighing (duplicates) five to seven unexposed filters on days of operation? Yes[ ] No[ ]

g. Describe what action is taken if the reweighed filters are not within ±0.02 mg of their original weights.

E. POST-RUN FILTER INSPECTION AND WEIGHING

1. Filters

a. Are exposed filters logged in for processing? Yes[ ] No[ ]

b. Are gloves worn during filter handling? Yes[ ] No[ ]

c. Are non-serrated forceps used to handle filters? Yes[ ] No[ ]

d. Are filters invalidated for:
   1) Total flow outside nominal 16.7 LPM (±10%) or coarse flow outside nominal 1.67 LPM (±10%) after altitude correction? Yes[ ] No[ ]
   2) Contamination or damage? Yes[ ] No[ ]
   3) Non-midnight start/stop times (±30 minutes)? Yes[ ] No[ ]
   4) Changes in flow rate of more than 10% from ideal operating flow rate? Yes[ ] No[ ]
   5) Changes in flow rate calibration of more than 10% as determined by field QC checks? Yes[ ] No[ ]
   6) Sampler not operating for 24 hours (±1 hour)? Yes[ ] No[ ]
   7) Missing or unobtainable information from the data sheet? Yes[ ] No[ ]

e. Briefly describe what action is taken if any of the above filter criteria are not met.

f. Are exposed filters stored after mass analysis? Yes[ ] No[ ]
If yes, where, under what conditions, and for how long?

E. POST-RUN FILTER INSPECTION AND WEIGHING (cont.)

2. Microbalance

   a. Are post-run microbalance checks performed as described in the pre-run filter inspection and weighing subsection? Yes[ ] No[ ]

   b. Are gross weights checked by reweighing five to seven exposed filters on days of operation (duplicates)? Yes[ ] No[ ]

3. Calculations

   a. Are readings corrected to standard conditions for calculations (temperature and pressure)? Yes[ ] No[ ]

   b. Give a brief description of the procedure and/or formula used to convert field data to final concentrations.

4. Quality Control (QC)

   a. As part of your QC program, what percent of filters are reweighed (duplicates)? _____%

   b. As part of your QC program, what percent of data are verified by recalculation?_____%

F. POST-RUN X-RAY FLUORESCENCE ELEMENTAL ANALYSIS

1. Filters

   a. For quantitative measurements on glass fiber or quartz filters, are filters analyzed for sulfur and lighter elements? N/A[ ] Yes[ ] No[ ]

   b. For analysis of quartz or glass fiber filters, are Mylar sheets used to cover the filters during analysis? N/A[ ] Yes[ ] No[ ]

   c. Are only filters with a minimum loading of 0.050 mg/cm2 analyzed? Yes[ ] No[ ]

   d. Are all large, loose particles on a filter deposit removed prior to analysis? Yes[ ] No[ ]

   e. Are flat-tipped stainless steel tweezers, cleaned with methanol, used for handling filters? Yes[ ] No[ ]

   f. Are latex gloves which have been wiped with a methanol-dampened Kimwipe used when handling filters? Yes[ ] No[ ]

POST-RUN X-RAY FLUORESCENCE ELEMENTAL ANALYSIS (cont.)

g. Is the work area cleaned with a methanol-dampened Kimwipe and covered prior to handling of filters? Yes[ ] No[ ]

h. Is each sample holder base and retainer ring wiped with a methanol-dampened Kimwipe prior to insertion of the filter? Yes[ ] No[ ]

I. Are filters checked for visual defects, large particles, filter damage, or other abnormalities? Yes[ ] No[ ]

j. Are filters with large amounts of potentially loose particles place between Mylar sheets during analysis? Yes[ ] No[ ]

2. XRF Analyzer

a. Is a performance check of the XRF analyzer conducted against a QA standard? Yes[ ] No[ ]

b. Is the QA standard performance check conducted every sampling run? Yes[ ] No[ ]

c. Does the QA standard performance check conducted with each run include results for 4 elements under specific conditions (tin (Sn) under condition 1, iron (Fe) under conditions 2 and 3, calcium (Ca) under conditions 3 and 4, and silicon (Si) under condition 5)? Yes[ ] No[ ]

d. What action is taken if the QA standard performance check results are not within $\pm$3% of true values?

3. Calculations

a. Are blank spectra developed? Yes[ ] No[ ]

b. Are blank spectra verified manually for unusual levels of contaminants or other abnormalities? Yes[ ] No[ ]

c. Is data validation performed on data for all sample analyses? Yes[ ] No[ ]

4. Quality Control (QC)

a. Are blanks from the same filter manufacturer/supplier in formulation of background/blank spectra included in each analyses? Yes[ ] No[ ]

F. POST-RUN X-RAY FLUORESCENCE ELEMENTAL ANALYSIS (cont.)

b. How many lab blanks (2 to 5 filters/lot) are analyzed?

_____

c. Are lab blanks analyzed in the first run for a given project? Yes[ ] No[ ]

d. As part of your QC program, what percent of filters are reanalyzed by XRF?_____%

e. What action is taken if the replicate (mg/cm2) data is not within ±10%, or within 3 times analytical uncertainties?

f. Are XRF Control Charts (graphical form of QA standard gross counts) generated each month? Yes[ ] No[ ]

g. Is a "Validation Summary" maintained? Yes[ ] No[ ]

h. What action is taken if any problems listed on "Validation Summary" cannot be resolved?

I. Is the accuracy performed by:

1) inter-laboratory comparisons on same sample set? Yes[ ] No[ ]
2) deviation of individual elements from a smooth curve of instrument response vs. atomic number? Yes[ ] No[ ]
3) gross counts of multi-element thin film standard? Yes[ ] No[ ]
4) analysis of NIST thin film standard? Yes[ ] No[ ]

G. REPORTING

1. Reporting

a. To whom are the results of the filter weighings reported? (e.g., EPA, ARB, etc.)_____

b. To whom are the results of the filter elemental analyses reported? (e.g., EPA, ARB, etc.)_____

c. To whom are the mass determinations specifically delivered? (Name of Division/Section and name of person)_____

d. How often are the results forwarded to the reporting organization? (e.g., quarterly, semi-annually, etc.)_____

e. In what format are the results reported? (e.g., hard copy, diskette, electronic)

_____

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APPENDIX AB.4.2

ANALYTICAL PROCESS SYSTEM EVALUATION

MONITORING AND LABORATORY DIVISION

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AB.4.2 ANALYTICAL PROCESS SYSTEM EVALUATION

AB.4.2.1 Microbalance - A performance audit of the microbalance used to weigh dichot filters requires the use of ASTM Class 1 standard weights. Since microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the performance evaluation of the filter weighing process be done in the following manner:

1. Review the maintenance and calibration log for each microbalance. Routine microbalance maintenance and calibrations must be performed by the manufacturers service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed one year.

2. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:

   a. Zero and calibration checks after every 10 or less filter weighings. These are microbalance internal checks and should be within ±0.004 mg at zero and ±0.002mg at 100 mg.

   b. Standard weight checks every day of the microbalance operation.

   c. Duplicate filter weighing for every 10 or less filters. Duplicate weights should be within ±0.02 mg of the tare (pre-run) filter weights.

   Note what action was taken if QC checks were out of limits.

3. Conduct a microbalance performance audit as follows:

   a. Select randomly and have the microbalance operator reweigh four equilibrated filters out of every group of 50 or less. For groups of 50 to 100, reweigh seven from each group. It is of primary importance to be sure that the sample is representative of the various conditions that may influence data quality.

   b. Record the original values and the audit weights on the audit form (Figure AB.4.1). Calculate the weight difference for each filter as follows:

      Difference = Original weight (mg) - Audit weight (mg)

      For unexposed filters, the difference should be less than ±20 micrograms (0.020 mg). For exposed filters, individual agencies may establish their own control limits, but the potential loss of volatile particles prohibits acceptance/rejection limits to be established by the U.S. EPA.

DICHOTOMOUS SAMPLING AND ANALYSIS PROGRAM PERFORMANCE AUDIT FORM

Agency_____

Address_____

Date of Site-Visit_____ Auditor(s)_____

1. Microbalance

   a. Observe technician weighing filters and look for the following activities:
      1) calibration checks at 0.0 mg and 100.0 mg Yes[ ] No[ ]
      2) logging of data Yes[ ] No[ ]
      3) check of RH and temperature Yes[ ] No[ ]
      4) filter handling technique (use of gloves and non-serrated forceps) Yes[ ] No[ ]
      5) weighing technique

   b. Duplicate weighing:
      (criteria: ±0.02 mg for pre-run filters)

ORIGINAL WEIGHT				
DUPLICATE WT.				

   c. Calibration Checks
      Model:_____ S/N:_____ Cal. Date:_____

      (10 mg)_____ (50 mg)_____ (80 mg)_____
      (100 mg)_____ (150 mg)_____ (criteria: ±0.001 mg)

2. Temperature and Relative Humidity Sensors

   a. Temperature sensor: Model_____ S/N_____ Cal. Date_____

   b. RH sensor: Model_____ S/N_____ Cal. Date_____

SENSOR AND LOCATION		ARB MEASUREMENT	District READING	CRITERIA
TEMPERATURE	ROOM			±1 °C
	CHAMBER			
RELATIVE HUMIDITY	ROOM			0.06
	CHAMBER			

Figure AB.4.2.1
Performance Audit Form

3. X-Ray Fluorescence Analyzer

Model:_____ S/N:_____ Cal. Date:_____

(criteria: no greater than ±3% difference)

ELEMENT	STANDARDS VALUES	DISTRICT RESPONSES	PERCENT DIFFERENCE

ARB standard:_____

Figure AB.4.2.1 (cont.)
Performance Audit Form

AB.4.2.2 X-Ray Fluorescence - A performance audit of the X-ray fluorescence (XRF) analyzer used to conduct elemental analysis of dichot filters requires the use of NIST standard filters (SRM 1832 or SRM 1833) or NIST-traceable standard filters. Since XRF analyzers are extremely delicate and complex instruments and should not be operated by inexperienced personnel, it is recommended that the performance evaluation of the filter elemental determination process be done in the following manner:

1. Review the maintenance and calibration log for each XRF analyzer. Routine XRF analyzer maintenance and calibrations may be performed by the operator; however, service maintenance, repairs, and major adjustments must be performed by the manufacturers service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed three months.

2. Review QC data records for the filter elemental determination analysis process. Ensure that the following QC activities have been performed and documented:

   a. Only filters with a minimum loading of 0.050 mg/cm^2 are analyzed. The optimum loading is approximately 0.150 mg/cm^2, or 1 mg/filter to 3 mg/filter, for 37 to 47mm sized filters.

   b. A quality assurance standard performance check must be included with each analytical run, and the results for four elements under specified analysis conditions must be presented, as follows:
      - tin (Sn) under condition 1
      - iron (Fe) under conditions 2 and 3
      - calcium (Ca) under conditions 3 and 4
      - silicon (Si) under condition 5

      The results of the QA standard analysis must be within ±3% of the true values.

      NOTE: The XRF analyzer operates at five excitation levels for analysis. Each excitation level is designated as a condition of the analyzer (conditions 1 through 5).

   c. The Multi-Channel Analyzer must be calibrated for the energy channels every instance in which the power was turned back on. The accuracy of the energy calibration must be within ±3eV based on Fe and Mo peaks in a Kevex stainless steel standard.

d. The XRF Analyzer should be calibrated using either elemental thin film or multiple element thin film standard. Calibration must be conducted every three months, when the QA standard performance check indicates a drift of more than ±3% in calibration, or when the results of the QA standard performance check consistently fall outside the ±5% intervals. The accuracy of the calibration is verified by analyzing a NIST thin film standard (SRM 1832 or SRM 1833).

e. Replicate filter analysis must be conducted at the rate of 10% of all samples analyzed for any given project. Replicate analysis results must be within ±10% of the original values (micrograms/cm^2), or within three times analytical uncertainties.

f. Data validation must be conducted and consists of:
- verifying all data entry is correct
- examining data results for internal consistency and reasonableness
- noting, for elements measured by two excitation conditions, the differences in concentrations must not be more than ±20% of the average concentration or no more than three times the sum of uncertainties
- nothing elevated concentrations of elements that are usually below the detection limit (Co, Ga, As, Se, Mo, Pd, Ag, Cd, In, Sn, Sb, Ba, La, Au, Hg, Ti, and U)
- evaluating data with respect to expected concentration ratios

g. When QC standard and replicate limits are exceeded, the recommendation is to reanalyze the sample. When calibration limits are exceeded, the recommendation is to recalibrate and then reanalyze the sample. If QC checks were out of limits, note what action was taken.

6. Record the XRF response values and the NIST standard certified values (NIST SRM 1832 or SRM 1833) on the audit form (Figure AB.4.1.1). Calculate the percent difference for each element as follows:

Difference = XRF response value (Counts) - Certified value

Percent Difference = (Difference/Certified value) x 100%

The percent difference should be no greater than ±3%. If the criteria are not met, then an AQDA should be issued.

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REFERENCES

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AA.5.0 REFERENCES

1. U.S. EPA Guidance Document 2.10, Monitoring PM10 in Ambient Air Using a Dichotomous Sampler, September 1997.

2. 40 CFR 50, Appendix J, July 1, 1996.

3. 40 CFR 53, July 1, 1996.

4. 40 CFR 58, July 1, 1996.

5. Air Monitoring Quality Assurance: Volume V, Audit Procedures Manual.

6. X-Ray Fluorescence (XRF) Analysis of Aerosol Filter Samples, Standard Operating Procedures, Desert Research Institute, December 1989.